

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : A23P 1/04, A23L 1/22		AI	(11) International Publication Number: WO 96/20612 (43) International Publication Date: 11 July 1996 (11.07.96)
(21) International Application Number: PCT/US95/16628		(81) Designated States: AL, AM, AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TT, UA, UG, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).	
(22) International Filing Date: 19 December 1995 (19.12.95)		Published <i>With international search report.</i>	
(30) Priority Data: 08/367,072 30 December 1994 (30.12.94) US			
(71) Applicant: TASTEMAKER [US/US]; 1199 Edison Drive, Cincinnati, OH 45216 (US).			
(72) Inventor: SOPER, Jon, C.; 6408 Luton Court, Huber Heights, OH 45424 (US).			
(74) Agents: JOSEPHIC, David, J. et al.; Wood, Herron & Evans, 2700 Carew Tower, Cincinnati, OH 45202 (US).			

RECORD COPY
CENTRAL FILE

(54) Title: **METHOD OF ENCAPSULATING FOOD OR FLAVOR PARTICLES USING WARM WATER FISH GELATIN, AND CAPSULES PRODUCED THEREFROM**

(57) Abstract

This invention is directed to a method of forming microencapsulated food or flavor capsules as well as the capsules produced by the method. The method includes providing food or flavor particles to be encapsulated, and forming a mixture of a warm water fish gelatin and the food or flavor particles in aqueous media. The method further includes microencapsulating the particles with the gelatin at elevated temperatures by complex coacervation to form the microencapsulated capsules. If desired, the method may further include the step of separating the capsules. In a preferred form, the method is conducted at a temperature of about 33 °C to about 35 °C. Preferably, the warm water fish gelatin used in the encapsulation method has a bloom of from about 150 to about 300 bloom, more preferably from about 250 to about 300 bloom. Many different kinds of food or flavor particles may be used, such as for example, vegetable oil, lemon oil, garlic flavor, apple flavor or black pepper. The invention also is directed to the food or flavor capsules produced by the method.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MW	Malawi
AT	Austria	GK	Georgia	MX	Mexico
AU	Australia	GN	Guinea	NE	Niger
BB	Barbados	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IZ	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KR	Kenya	RO	Romania
BY	Belarus	KG	Kyrgyzstan	RU	Russia Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	KZ	Kazakhstan	SG	Singapore
CH	Switzerland	LJ	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SZ	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Latvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

-1-

**METHOD OF ENCAPSULATING FOOD OR FLAVOR PARTICLES
USING WARM WATER FISH GELATIN, AND CAPSULES
PRODUCED THEREFROM**

Background of the Invention

This invention relates to microencapsulation of food or flavor particles, and more particularly to microencapsulation of food or flavor particles by complex coacervation at elevated temperatures using a warm water fish gelatin.

U.S. Patent No. 5,035,896 to Apfel et al. teaches water insoluble drugs coated by simple coacervation using a low bloom fish gelatin which coacervates at ambient temperature, i.e., room temperature of about 16°-27°C. However, the '896 patent does not disclose the use of fish gelatin in encapsulating other kinds of particles. Furthermore, the '896 coacervation process is limited to ambient or room temperature coacervation.

Summary of the Invention

This invention is directed to a method of forming

-2-

microencapsulated food or flavor capsules as well as the capsules produced by the method. The method includes providing food or flavor particles to be encapsulated, and forming a mixture of a warm water fish gelatin and the food or flavor particles in aqueous media.

5 The method further includes microencapsulating the particles with the gelatin at elevated temperatures by complex coacervation to form the microencapsulated capsules. If desired, the method may further include the step of separating the capsules.

In a preferred form, the method is conducted at a
10 temperature of about 33°C to about 35°C. Preferably, the warm
water fish gelatin used in the encapsulation method has a bloom of
from about 150 to about 300 bloom, more preferably from about
250 to about 300 bloom. Many different kinds of food or flavor
particles may be used, such as for example, vegetable oil, lemon oil,
15 garlic flavor, apple flavor or black pepper. The invention also is
directed to the food or flavor capsules produced by the method.

This invention offers several benefits and advantages.
For example, the method provides a way of encapsulating food or
flavor particles using fish gelatin instead of other gelatin such as
20 bovine or porcine gelatin. Also, the fish gelatin employed is a kosher
fish gelatin; therefore, any kosher food or flavor particle will remain
kosher upon encapsulation. In addition, the coacervation method is
conducted at elevated temperatures, and does not require
ambient/room temperature for coacervation.

-3-

Detailed Description of the Invention

The invention is directed to a method of forming microencapsulated food or flavor capsules, as well as to the capsules produced from the method. The method includes providing food or flavor particles to be encapsulated, and forming a mixture of a warm water fish gelatin and the food or flavor particles in an aqueous media. The method further includes microencapsulating the particles with the gelatin at elevated temperatures by complex coacervation to form the microencapsulated capsules.

The warm water fish gelatin used in the method may be any warm water fish gelatin. However, in a preferred form of the invention, the fish gelatin has a bloom of from about 150 to about 300 bloom, and more preferably, a bloom of from about 250 to about 300 bloom. Such high bloom warm water fish gelatins are available from Food Industries Testing of Miami, Florida under the product name Gelcine KFX. One of the advantages of using the fish gelatin is that it is kosher. Therefore, kosher food or flavor particles retain their kosher status upon encapsulation with the fish gelatin.

As stated above, the method further includes microencapsulating the particles with the gelatin at elevated temperatures by complex coacervation. As used herein, "elevated temperatures" means temperatures above ambient or room temperatures, i.e., above 16°-27°C. During complex coacervation, a protein coating is formed over the food or flavor particles to produce

-4-

microencapsulated capsules. The protein coating may then be cross linked by covalent or ionic bonding with a cross linking agent, and if desired, the capsules may be spray dried or otherwise dewatered at a suitable temperature for the removal of water.

5 By employing the method of this invention, food or flavor particles in amounts of up to 95% by weight, typically within the range of about 50% to 95% by weight, are encapsulated in a protein coating, i.e., at a ratio of about 10:1 to about 5:1 of particles to coating. Usually, about 70% to about 95% by weight particle encapsulation is achieved. In another feature of the 10 invention, a drying aid is added to the coacervated emulsion of food or flavor particles before spray drying or dewatering. This drying aid has the effect of lubricating these spray dried, or otherwise dewatered particles and provides for uniform distribution of the 15 particles.

A variety of food or flavor particles may be encapsulated, such as for example, vegetable oil, lemon oil, garlic flavor, apple flavor or black pepper. Examples of other flavor oils include flavoring aromatic compounds and/or oils, oleoresins and extracts derived from plants, leaves, flowers, fruits and so forth, and combinations thereof. Flavor oils include cinnamon oil, oil of wintergreen, peppermint oil, bay oil, thyme oil, spearmint oil, cassia oil, and the like. Artificial, natural and synthetic fruit flavors such as vanilla, and citrus oils including lemon, orange, grapefruit, lime, and

-5-

fruit essences including apple, pear, peach, strawberry, cherry, and so forth, may be used. These flavoring oils may be used individually or in a mixture as is well known in the art.

The coating layer, as prepared by complex coacervation
5 comprises colloidal materials which must be hydrophilic, gellable and ionizable. Colloidal materials may be selected from the group consisting of a warm water fish gelatin, alginates, casein, gum arabic, carboxymethylcellulose and the like, and mixtures thereof.

Complex coacervation is practiced usually by first
10 forming a solution of a first colloidal material, such as the warm water fish gelatin, in water above its gelation temperature.
Separately, a second colloidal material, such as gum arabic or carboxymethylcellulose for example, is added to water to form a clear solution. The two solutions then are mixed, the temperature
15 may be reduced, and the flavor oil is mixed into the resulting solution at a mixing speed to form the desired emulsion. Complex coacervation, or aggregation to uniformly distribute the colloidal materials around the food or flavor particles, then is carried out by diluting the emulsion with water, or adjusting the pH and allowing an
20 elapsed time for the colloid to coat the particles. Then, in order to render the gelatin capsules insoluble, it is usually necessary to cross link the colloidal coating on the particles, and in a preferred form, glutaraldehyde is used to cross link the gelatin coating surrounding the particles. Alum also may be used to cross link the coated food

-6-

or flavor particles.

It is preferred to add a drying aid to the slurry of coated microcapsules in aqueous medium prior to spray-drying or dewatering. Silicon dioxide is a preferred drying agent having a particle size of less than 400 mesh, but the fineness of the drying agent is not critical. The coated flavor capsules are now ready for spray-drying and this may be accomplished by employing a spray-drying tower fitted with an air atomization nozzle for spraying countercurrent to the heated airflow. The coated food or flavor capsules are delivered to the spray nozzle by a pump at a suitable rate per hour. Atomization may be achieved in a number of ways. For example, air atomization, spinning disk or airless atomization may be employed.

The resulting food or flavored capsules may be prepared in different forms, such as a liquid suspension or slurry (typically about 1% to about 40% by weight solids), a paste (about 36% to about 60% by weight solids), or a dry powder. The slurry form may have advantage in certain applications because of the benefits of particle cost savings, easy handling, and better particle containment because of reduced particle volatility in the water. The aqueous food or flavor capsules may be delivered by a pump at a suitable rate per hour in processing to afford easy handling.

The following examples illustrate the practice of this invention. It will be understood, however, that these examples are

-7-

not intended to limit the scope of the invention, which is to be determined by the appended claims and their equivalents.

Example 1

5 Aqueous Capsules of Flavor Particles and Cross-Linked Warm Water Fish Gelatin Coating

An aqueous suspension of vegetable oil microcapsules was produced according to the method of the invention. The warm water fish gelatin 300 bloom Gelcine KFX supplied by Food Industry Testing of Miami, Florida was used to form 200 grams of a 10% fish 10 gelatin solution at 33°C. Then 100 grams of a 4% CMC/ARABIC gum solution was prepared at room temperature. The gum and gelatin solutions were combined followed by the addition of 160 grams of vegetable oil. 600 grams of dilution water at 35°C was added to the emulsion, and the resulting solution was cooled over a 15 30-minute period to a temperature of about 25°C. 2.5 grams of a 50% aqueous solution of gluteraldehyde was added in order to cross link the gelatin. The result was an excellent aqueous suspension of microcapsules. In addition, a similar batch of capsules were prepared using the above steps, but substituting a 250 bloom warm 20 water fish gelatin for the 300 bloom gelatin used above.

Additional aqueous suspensions of microcapsules were prepared using lemon oil, garlic flavor or apple flavor instead of the vegetable oil discussed above. The method conducted using these particular flavors also resulted in an excellent aqueous suspension of 25 microcapsules.

Example 2**Spray-Dried, Free-Flowing Capsules of Flavor Particles And Cross-Linked Warm Water Fish Gelatin Coating**

The aqueous suspensions of lemon oil, garlic flavor and
5 apple flavor capsules prepared in Example 1 above were further
processed to form dry powders of flavor capsules. In order to form
the dry powders, silicon dioxide was mixed into the slurries or
aqueous suspensions to act as a drying aid upon spray-drying. The
suspensions were then spray-dried using the spray-drying technique
10 discussed above. This process resulted in dry, free flowing
microcapsules.

-9-

I CLAIM:

1. A method of forming microencapsulated food or flavor capsules comprising the steps of:

providing food or flavor particles to be encapsulated;

5 forming a mixture of a warm water fish gelatin and said food or flavor particles in aqueous media; and microencapsulating said particles with said gelatin at elevated temperatures by complex coacervation to form said microencapsulated capsules.

10 2. The method of claim 1 conducted at a temperature of about 33°C to about 35°C.

3. The method of claim 1 further comprising the step of separating said capsules.

15 4. The method of claim 1 wherein said fish gelatin has a bloom of from about 150 to about 300 bloom.

5. The method of claim 3 wherein said fish gelatin has a bloom of from about 250 to about 300 bloom.

-10-

6. The method of claim 1 wherein said particles to be
encapsulated are selected from the group consisting of vegetable oil,
lemon oil, garlic flavor, apple flavor and black pepper.

7. The product produced by the method of claim 1.

5 8. The product produced by the method of claim 2.

9. The product produced by the method of claim 3.

10. The product produced by the method of claim 6.

11. The product produced by the method of claim 1
wherein said product is kosher.

-11-

12. A method of forming microencapsulated liquid flavor capsules comprising the steps of:

providing liquid flavor particles to be encapsulated;

forming a mixture of a warm water fish gelatin and said

5 liquid flavor particles in aqueous media; and

microencapsulating said particles with said gelatin at

elevated temperatures by complex coacervation to form said

microencapsulated capsules.

INTERNATIONAL SEARCH REPORT

International Application No PCT/US 95/16628

A. CLASSIFICATION OF SUBJECT MATTER IPC 6 A23P1/04 A23L1/22	
--	--

According to International Patent Classification (IPC) or to both national classification and IPC	
---	--

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols) IPC 6 A23P A23L A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
--

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO,A,93 19621 (TASTEMAKER) 14 October 1993 see the whole document ---	1-11
Y	EP,A,0 347 751 (F.HOFFMAN-LA ROCHE) 27 December 1989 see column 1, line 32 - column 3, line 20 see examples 1,3,5-9 ---	1-11
Y	EP,A,0 273 823 (MERO ROUSSELOT SATIA) 6 July 1988 see the whole document ---	1-11
Y	EP,A,0 346 879 (WARNER-LAMBERT) 20 December 1989 see the whole document & US,A,5 035 896 cited in the application ---	1-11
	-/-	

<input checked="" type="checkbox"/> Further documents are listed in the continuation of box C.
--

<input checked="" type="checkbox"/> Patent family members are listed in annex.
--

* Special categories of cited documents :

- *'A' document defining the general state of the art which is not considered to be of particular relevance
- *'B' earlier document but published on or after the international filing date
- *'L' document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *'O' document referring to an oral disclosure, use, exhibition or other means
- *'P' document published prior to the international filing date but later than the priority date claimed

*'T' later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

*'X' document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

*'Y' document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

*'A' document member of the same patent family

1

Date of the actual completion of the international search

Date of mailing of the international search report

12 April 1996

26.04.1996

Name and mailing address of the ISA

Authorized officer

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+ 31-70) 340-2040, Tel. 31 651 epc nl.
Fax: (+ 31-70) 340-3016

Vuillamy, V

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 95/16628

C(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	P.B.DEASY: "Microencapsulation and Related Drug Process" 1984 , M.DEKKER , NEW YORK - USA XP002000439 pp.64-75 see page 69 -----	2
A	WO,A,91 17821 (DANOCHEMO) 28 November 1991 see the whole document -----	

1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 95/16628

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
W0-A-9319621	14-10-93	AT-T-	129855	15-11-95
		AU-B-	3969893	08-11-93
		CA-A-	2130442	14-10-93
		CZ-A-	9402365	17-05-95
		DE-D-	69300768	14-12-95
		EP-A-	0633732	18-01-95
		FI-A-	944517	29-09-94
		JP-T-	8501441	20-02-96
		SK-A-	114994	05-01-95
<hr/>				
EP-A-347751	27-12-89	AU-B-	623908	28-05-92
		AU-B-	3657389	04-01-90
		HK-A-	142595	15-09-95
		IE-B-	62542	08-02-95
		JP-A-	2051594	21-02-90
		NO-B-	177374	29-05-95
		PT-B-	90948	30-11-94
		US-A-	5478569	26-12-95
<hr/>				
EP-A-273823	06-07-88	FR-A-	2608456	24-06-88
		CA-A-	1323307	19-10-93
		JP-A-	63197540	16-08-88
		US-A-	5051304	24-09-91
<hr/>				
EP-A-346879	20-12-89	US-A-	5035896	30-07-91
<hr/>				
W0-A-9117821	28-11-91	AU-B-	7903191	10-12-91
		EP-A-	0530277	10-03-93
<hr/>				

PATENTS SUMMARY

11/06/98

Page 1

Country : Patent Cooperation Treaty (PCT)

Patent #: 96-020612 A

Index#: 5561

Inventor: Jon C. Soper

Issue Date: 07/11/96

Assignee: Tastemaker

Date of Application: 12/30/94

Title:

METHOD OF ENCAPSULATING FOOD OR FLAVOR PARTICLES USING WARM
WATER FISH

GELATIN, AND CAPSULES PRODUCED THEREFROM

Desc.:

A method of forming microencapsulated food or flavor capsules and the products so produced are claimed. A mixture of warm water fish gelatin and the food or flavor particles to be encapsulated is prepared in an aqueous media. The microcapsules are formed at evaluated temperature by complex coacervation. The product may be spray-dried to obtain free-flowing microcapsules. The product is kosher. Claimed core materials are vegetable oil, lemon oil, garlic flavor, apple flavor and black pepper.

Key Words:

400 FLAVORS/FRAGRANCES

409 Other Spice

411 Fruit

466 Spices/Herbs

472 Encapsulants

474 Gelatin (Gels)

491 Chemicals

521 Microencapsulation/Spray Drying/Freeze Drying

569 Improved or Easier Processing

799 Other Company/Institution

810 Patent Cooperation Treaty (PCT)